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ENVIRONMENTAL PROTECTION OF PYROTECHNIC PRODUCTS AND DEVICES.(U)
JUL 74 W BRENNER, J J TIMLIN DAAA21-73-C-0117

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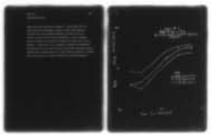

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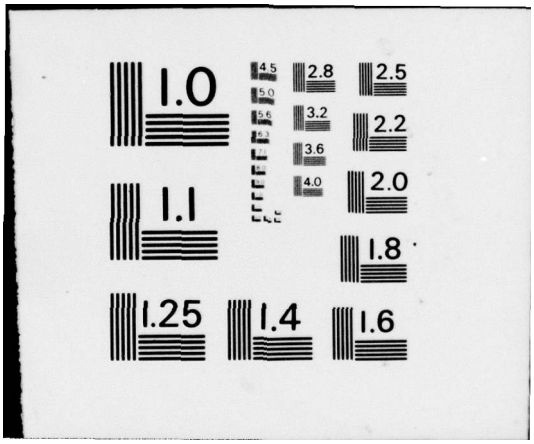
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Report No. 4

(6) ENVIRONMENTAL PROTECTION OF PYROTECHNIC PRODUCTS AND DEVICES.

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James J. Timlin

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Picatinny Arsenal

Dover, New Jersey 07801

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Polytechnic Institute of New York

Brooklyn, New York 11201

257350 LB

Report #4

Pyrotechnic Products

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Authors of reports were formerly associated with N.Y.U. When they left & went to Polytechnic Institute of N.Y. they continued to work on contract.
per J. Norman.

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Report #4

Pyrotechnic Products

This is the fourth progress report under contract DAAA 21-73-C-0017 commenced at New York University on December 18, 1972 and covers the work period starting June 8, 1973 and ending July 1, 1974. The principal objective of these experimental investigations are the development of more effective water-proofing techniques for reactive powders such as aluminum and magnesium as well as sodium nitrate, black powder, etc. without detracting from their performance characteristics as a component of flares and similar devices. Another objective is to improve the liquid water and water vapor resistance of cartridge cloth bags. The most important specific task is to develop protective treatments and/or coatings as well as appropriate application procedures for use with these reactive powders which will enhance their long term service capabilities in the presence of moisture, elevated temperatures and other adverse environmental conditions likely to be encountered during manufacture or upon prolonged storage. The cloth cartridge bags must be protected against analogous adverse environmental conditions in order to optimize the ballistics of non-metallic cartridge cases upon combustion.

A very extensive program of research and development work has been initiated and is being completed in order to develop environmental protective coating systems as well as application methods for magnesium and aluminum metal powders, sodium nitrate, black powder and also various cartridge cloth materials. Emphasis was placed on formulations and application technology capable of yielding reproducible results in terms of quantitative measurements of liquid water and water vapor resistance. Equipment has been described in previous reports of this series.

Report #4

Pyrotechnic Products

Selected experimental techniques were further explored for the application of hydrophobic polymeric coatings primarily from organic solutions especially onto magnesium powders of known particle size in order to improve upon their environmental resistance against both water vapor and liquid water, at minimum add on's. The use of different types and degrees of agitation during the drying of solution coated magnesium powders was carefully investigated for the purposes of both optimizing the uniformity of the polymeric coatings and also minimize agglomeration of the coated metal particles. Analogous variables were investigated for black powder particles whose hygroscopicity has long been a problem in many uses.

The results of past and currently carried out experimental studies emphasized again the primary importance of the method of application for obtaining reproducible water resistance data with various reactive metal powders and also black powder particles. Experiments have demonstrated the utility of the previously described fluidized bed which was designed and constructed in the summer of 1973 in these laboratories and has been in operation since then. This fluidized bed has given very satisfactory performance and design and construction of a larger unit is being contemplated for water-proofing greater amounts of reactive metal powders, etc.

Evaluation of the quality of the polymeric protection layer on magnesium powder particles was accomplished as discussed earlier by contacting the polymer coated magnesium metal powder particles with sodium nitrate and measured small amounts of water. This test procedure was decided upon to

approximate as closely as possible actual anticipated use conditions and has been revised previously. It can be carried out in a number of ways. In one typical series of tests 50 grams of treated magnesium powder particles were mixed with 50 grams of sodium nitrate (anhydrous) and one gram of water and placed in stainless steel aerosol containers provided with a pressure gauge. The pressure changes were then measured after various time periods at previously determined temperatures. Another technique which is being used involves the measurement of pressure changes by means of an inclined manometer setup attached to a container for these ingredients.

During the time period covered by this report various samples of magnesium aluminum and sodium nitrate coated with microcrystalline/paraffin/Elvax formulations, VAR, PVC compositions from solvent solutions containing various active materials were prepared and sent to the Picatinny Arsenal for evaluation. Altogether over 35 lbs. of coated powders were supplied to the Arsenal for their work.

I. Reactivity of various untreated metallic powders

Table #1 summarizes experimentally derived data which indicate the relative reactivity of four different metallic powders, i.e. Alcoa 8μ aluminum, Alcoa 6μ aluminum, 20/50 mesh magnesium, and 200/325 magnesium all of which were supplied by the Picatinny Arsenal for coating and subsequent evaluation. They were tested by the previously described sodium nitrate additive method and represent the average of three runs.

Table #1. Reactivity of untreated metallic powder/sodium nitrate compositions with liquid water. (50 gms untreated metallic powder, 50 gms sodium nitrate, 1 gm water).

(dp/dt) reaction rates [mm/sec x 197]

Time (seconds)	6 μ Aluminum	8 μ Aluminum	20/50 Magnesium	200/325 Magnesium
115	.063	.064	.027	.126
225	.061	.056	.021	.053
350	.050	.038	.015	.044
465	.057	-	.010	.038
575	.036	.040	.007	.028
690	.026	.038	.005	.026
805	.019	.018	.008	.022
920	-	.016	-	.015
1035	-	.015	-	.013
1150	-	.013	-	-
1265	-	0.10	-	.006

Table #2 contains pressure build-up versus exposure time data for various metallic powder samples kept at 125°F, time periods ranging from four to twenty-four hours. Data represent the average of three runs.

Table #2. Pressure build-up vs time data for various untreated metallic powder/$NaNO_3$ liquid water compositions. (Temperature = 125°F) (50 gms untreated metallic powder, 50 gms $NaNO_3$, 1 gm H_2O).

	Pressure generation (mm of Hg/hr.) 4 hrs.	24 hrs.
6 μ Aluminum	7.2	6.9
8 μ Aluminum	7.0	6.8
20/50 mesh magnesium	1.8	1.2
200/325 mesh magnesium	0.9	1.1

These test results clearly show the greater pressure generation of the aluminum powders tested as against the magnesium powders evaluated.

II. Reactivity of magnesium powder coated with various Microcrystalline/Paraffin/Elvax 60/40/1 coating compositions and Silicones.

Magnesium powder samples were spray coated and dried with a wax coating system of:

99 gms 60% microcrystalline/40% paraffin wax/1 gm Elvax

1900 gms benzene

The coating experiments were carried out at 180°F and conditions were varied in such a manner so that five different coating weight pickups could be obtained. Also, magnesium was sprayed and dried within the fluidized

bed with Dow Corning Silicone C-2-0563 used as a 10% solution in trichloroethylene. Table #3 shows data showing the reactivity of these compositions when tested at room temperature conditions. Data represent the average of three runs.

Table #3. Reactivity of treated magnesium metal powder/sodium nitrate composition with liquid water. (50 gms treated magnesium powder, 50 gms sodium nitrate, 1 gm water).

(dp/dt) reaction rates [mm/sec x 197]

	Coating wt. pick-up					
	Microcrystalline Paraffin/Elvax blend (60/40/1)					Silicone C-2-0563
Time in seconds	%	0.635%	0.800%	1.51%	3.19%	1.6%
125	0.056	0.044	0.041	0.037	0.047	0.053
240	0.012	0.013	0.010	0.005	0.019	0.004
355	0.011	0.010	0.006	0.003	0.009	0.004
470	0.011	0.008	0.004	0.001	0.006	0.002
585	0.010	0.004	0.002	0.001	0.007	0.002
700	0.010	0.005	0.001	0.001	0.007	-
815	0.009	0.003	0.002	-	0.006	-
930	0.008	-	0.003	-	0.004	-
1045	0.007	0.001	-	-	0.003	-
1160	-	0.001	-	-	0.001	-
1275	-	-	-	-	0.001	-

In Table #4 there are summarized pressure build-up vs. time data for treated magnesium metal powder samples held at 125°F for time periods ranging from 4 to 24 hours. These data represent the average of three runs each.

Table #4. Pressure build-up vs exposure time data for various treated magnesium metal powder samples in compositions containing magnesium metal powder/ sodium nitrate/liquid water at 125°F. (50 gms treated magnesium powder, 50 gms sodium nitrate, 1 gm water).

Coating Composition	Wt. % pick-up	Pressure generation (mm Hg/hour)	
		4 hrs.	24 hrs.
Microcrystalline/Paraffin/Elvax; 60/40/1	0	6.8	6.1
Microcrystalline/Paraffin/Elvax; 60/40/1	0.635	6.0	6.1
Microcrystalline/Paraffin/Elvax; 60/40/1	0.800	5.7	5.0
Microcrystalline/Paraffin/Elvax; 60/40/1	1.51	5.7	4.8
Microcrystalline/Paraffin/Elvax; 60/40/1	3.19	6.1	6.0
Silicone C-2-0563; 60/40/1	1.60	6.4	6.2

III. Black Powder

Black powder supplied by Picatinny Arsenal was coated in the previously described ball mill by treating it with a solution of Mobilwax 2300 microcrystalline wax, paraffin wax and three different grades of Elvax in benzene (60 microcrystalline wax, 40 paraffin wax, 1 Elvax). The three grades of Elvax

ethylene vinyl acetate copolymer were Elvax 40, 250 and 350. These grades differed in ethylene polymer content, etc. The treatment procedure has been described previously.

The thusly coated samples were then exposed to 74.7% humidity for 1 to 4 day time periods at ambient temperatures. The percentage weight gain was determined as an indication of the powder's inertness to liquid water. Table #5 summarizes the results of these experiments as a function of the Elvax binder type.

Table #5. Water absorption properties of black powder coated with Microcrystalline wax/Paraffin wax/Elvax compositions for environmental protection as a function of Elvax binder type.

	% Wt. pickup of treated black powder samples after			
	one day	two days	three days	four days
Control	5.18	6.34	7.62	7.98
Elvax 40	2.64	3.08	3.57	4.06
Elvax 250	2.44	3.07	3.62	4.21
Elvax 350	0.51	0.51	0.51	0.57

The test data show Elvax 350 to be the preferred binder for this protective treatment composition.

IV. Samples forwarded to Picatinny Arsenal

Selected reactive substrates were coated in the fluidized bed with two different candidate environmental protection systems and forwarded to Picatinny Arsenal for their evaluation studies. The specific samples of such reactive substrates were magnesium metal powder, sodium nitrate and also 50/50 mixtures of magnesium metal powder and sodium nitrate, all supplied by Picatinny Arsenal. The protective coating compositions used were:

Pyrotechnic Products

1) Wax/Elvax i.e. 50% mixture of 60/40 Mobil Microcrystalline Wax/ Paraffin Wax; 0.5% Elvax 350 and 99.45% toluene; called "Wax".

2) "VAAR" i.e. 5% Var, 95% methanol (preferred over butyl acetate on account of its lower boiling point); called "VAAR".

In total twelve samples were furnished to Picatinny Arsenal as summarized in detail below in Table #6.

Table #6. Environmental Protection Systems and substrates used for samples forwarded to Picatinny Arsenal evaluation studies.

Sample #	Wt. % coating pickup	Type of coating	Type of substrate
1	2.5	"Wax"	magnesium powder
2	2.5	"Wax"	sodium nitrate
3	2.5	"Wax"	50/50 magnesium powder/ sodium nitrate
4	5.0	"Wax"	magnesium powder
5	5.0	"Wax"	sodium nitrate
6	5.0	"Wax"	50/50 magnesium powder/ sodium nitrate
7	2.5	"VAAR"	magnesium powder
8	2.5	"VAAR"	sodium nitrate
9	2.5	"VAAR"	50/50 magnesium powder/ sodium nitrate
10	5.0	"VAAR"	magnesium powder
11	5.0	"VAAR"	sodium nitrate
12	5.0	"VAAR"	50/50 magnesium powder/ sodium nitrate

The following coating procedure was employed for preparation of these samples. 1000 gms of substrate were weighed and charged into the fluidized bed drier. Appropriate amounts of coating compositions for environmental protection of the substrates were then added as volatile organic solvent solutions. The drier blower was turned on and the air pressure was set so as to cause a sufficient amount of agitation of the substrate particles for dispersion and mixing while the volatile solvent was being evaporated. The sample was kept mixing in the fluidized bed until all the solvent had been removed and the substrate particles were dry. This was determined by weight measurements.

V. Samples tested for water absorption

Picatinny Arsenal supplied various samples consisting of pressed composites of magnesium metal powder and sodium nitrate, which had been previously treated for improved environmental protection for water absorption testing at 75% RH. These tests are summarized below in Table #7.

Table #7. Water absorption properties (75% RH) of variously treated pressed magnesium metal powder/sodium nitrate composites.
(Samples submitted by Picatinny Arsenal)

Type and amount of environmental protection system vs weight change (water absorption)

Time, minutes	2.5% "Wax"	5.0% "Wax"	2.5% "VAAR"
185	0.041	0.0038	0.0087
355	0.0030	0.0045	0.0075
1475	0.0178	0.0168	0.0246
2885	0.0201	0.0259	0.0313
4295	0.0214	0.0290	0.0335

These results are shown also in Figure #1. The data show that the "Wax" system is substantially superior to VAAR at equal add on's. They also show that increasing (doubling) the amount of the Wax protective coating did not improve performance. This is important because low add on's are obviously desirable for optimal use of these materials. Additional R & D is suggested to optimize the formulation and application of these novel improved environmental protection systems, particularly as regards achieving a) further add on reductions; b) greater water resistance and c) enhanced durability and scratch resistance.

Figure No. 1
Weight Change vs. Time
for Environmental protected Mg/$NaNO_3$
composites